

Tribological properties of NiAl-based composites containing Ag_3VO_4 nanoparticles at elevated temperatures

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ABSTRACT

In order to improve the tribological properties of NiAl intermetallic, Ag_3VO_4 nanoparticles with a size of 100–200 nm were synthesized by hydrothermal method and NiAl/ Ag_3VO_4 composites were successfully prepared by mechanical alloying and sintering technique. The composition and microstructure of milled powders and sintered composites were characterized and the tribological properties were evaluated. The results showed that NiAl-based composites consisted of B2 ordered NiAl matrix and metallic Ag/vanadium oxide precipitates, which were formed by the decomposition of silver vanadate. Wear testing results indicated that NiAl-based composites containing silver vanadate exhibited superior tribological properties at elevated temperatures. Subsequently, Raman results demonstrated that the reproduction of silver vanadate was responsible for the improvement of tribological properties at elevated temperatures.

Keywords:

NiAl/ Ag_3VO_4 composites
Tribological properties
Elevated temperatures
Lubrication mechanism

1. Introduction

NiAl intermetallic is the most promising candidate for elevated temperatures structural applications due to its attractive properties, such as high specific strength, high melting point, good thermal conductivity and excellent high temperature oxidation resistance [1–5]. Those excellent performances make NiAl intermetallic becomes a potential material in relative sliding components, such as turbochargers, hydroturbines, cutting tools and turbines. Thus, poor tribological properties of NiAl intermetallic at elevated temperatures are the drawback of engineering application [6–8]. Hawk's work indicates that the room temperature wear-resistance of NiAl intermetallic is more better than Fe_3Al , TiAl, Al_2O_3 ceramics and PS-ZrO₂ ceramics when test against SiC [9]. Nevertheless, severe adhesive wear induces NiAl intermetallic quick failure at elevated temperatures [10]. It is well known that addition of suitable solid lubricants might be an effective way to improve the tribological properties. Therefore, how to find suitable lubricants for NiAl intermetallic is the primary work to be studied.

In the last decade, silver vanadate (Ag_3VO_4) attracts many researchers' concentration in an effort to develop efficient, visible

light-driven photocatalysts [11–13]. More recently, silver vanadate becomes an intense focus in tribology because of the strong potential application for lubricating at elevated temperatures [14,15]. It is reported that VN/Ag adaptive coatings show lower friction and wear rate when system temperature exceed 500 °C and the further studies indicate that the formation of lubricious Ag_3VO_4 and AgVO_3 film on the worn surface is beneficial for the improvements of tribological properties [16]. To investigate the lubricating effect of silver vanadate as solid lubricants, pure Ag_3VO_4 and AgVO_3 powders are produced by hydrothermal method and then the high temperatures tribological properties are studied [17,18]. First, monoclinic structure Ag_3VO_4 powders are burnished to VN coating, and then the tribological properties of VN/ Ag_3VO_4 coating are tested by ball-on-disk tribometer. The results show that the friction coefficient of VN/ Ag_3VO_4 coatings is significantly decreased at elevated temperatures, which can lower to 0.2 at 750 °C [17]. Furthermore, in the study of D.P Singh, AgVO_3 powders are burnished to H13 steel disk and the tribological properties are tested against Si_3N_4 ball at elevated temperatures. The wear testing results indicate that the friction coefficient of AgVO_3 lubricant is reduced to 0.2 at 700 °C, which may be due to the fact that a self-lubricating layer containing V_2O_5 , Ag_3VO_4 and AgVO_3 are formed on the worn surface during high temperatures wear testing [18]. Thus, it can be deduced that silver vanadate may also be a good high temperatures lubricant for NiAl intermetallic. To our current knowledge, no study has been performed on the tribological properties of NiAl/ Ag_3VO_4 composites.

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Therefore, effect of silver vanadate (Ag_3VO_4 nanoparticles) on the tribological properties of NiAl intermetallic at elevated temperatures is studied in this work.

The goal of this research was to prepare NiAl/ Ag_3VO_4 composites with excellent tribological properties at elevated temperatures. In previous works, nanocrystalline NiAl intermetallic has been successfully synthesized in our lab [10,19]. Thus, NiAl/ Ag_3VO_4 composites were designed based on the hypothesis that nanocrystalline NiAl offers good mechanical properties and Ag_3VO_4 could be the high temperatures lubricants [20]. The tribological properties of NiAl/ Ag_3VO_4 composites were tested from room temperature to 900 °C and NiAl intermetallic with no silver vanadate addition were prepared and tested under the same conditions.

Table 1
The density and microhardness of sintered NiAl-based composites.

Composites	Composition (wt%)		Microhardness	Density (g/cm^3)
	Ni-50 at% Al	Ag_3VO_4		
NA	100	0	549.69	5.69
NA5V	95	5	487.53	5.88
NA10V	90	10	426.37	6.03
NA15V	85	15	384.03	6.21

2. Experimental details

2.1. Synthesis of silver vanadate nanoparticles

Silver vanadate (Ag_3VO_4) nanoparticles were successfully synthesized by hydrothermal method [17]. Commercially available vanadium oxide (V_2O_5), silver nitrate (AgNO_3), and sodium hydroxide (NaOH) reagents were used as received without any further purification. First, 100 ml of 0.02 M V_2O_5 solution was prepared firstly, and then 100 ml of 0.12 M NaOH solution was slowly added to V_2O_5 solution under magnetic stirring conditions at RT. Once a clear yellow solution was achieved, then the 100 ml 0.06 M AgNO_3 was added to the yellow solution drop by drop. The mixture solution was settled at room temperature for 72 h and the liquid was poured off to recover the orange precipitates, and then the precipitates were washed with distilled water to remove the ions (possibly remaining in the product) and finally dried under ambient conditions.

2.2. Preparation of NiAl nanocomposites

NiAl/ Ag_3VO_4 composites were successfully prepared by mechanical alloying (MA) and hot-pressing sintering technique. First, commercially available powders of nickel, aluminum were mixed

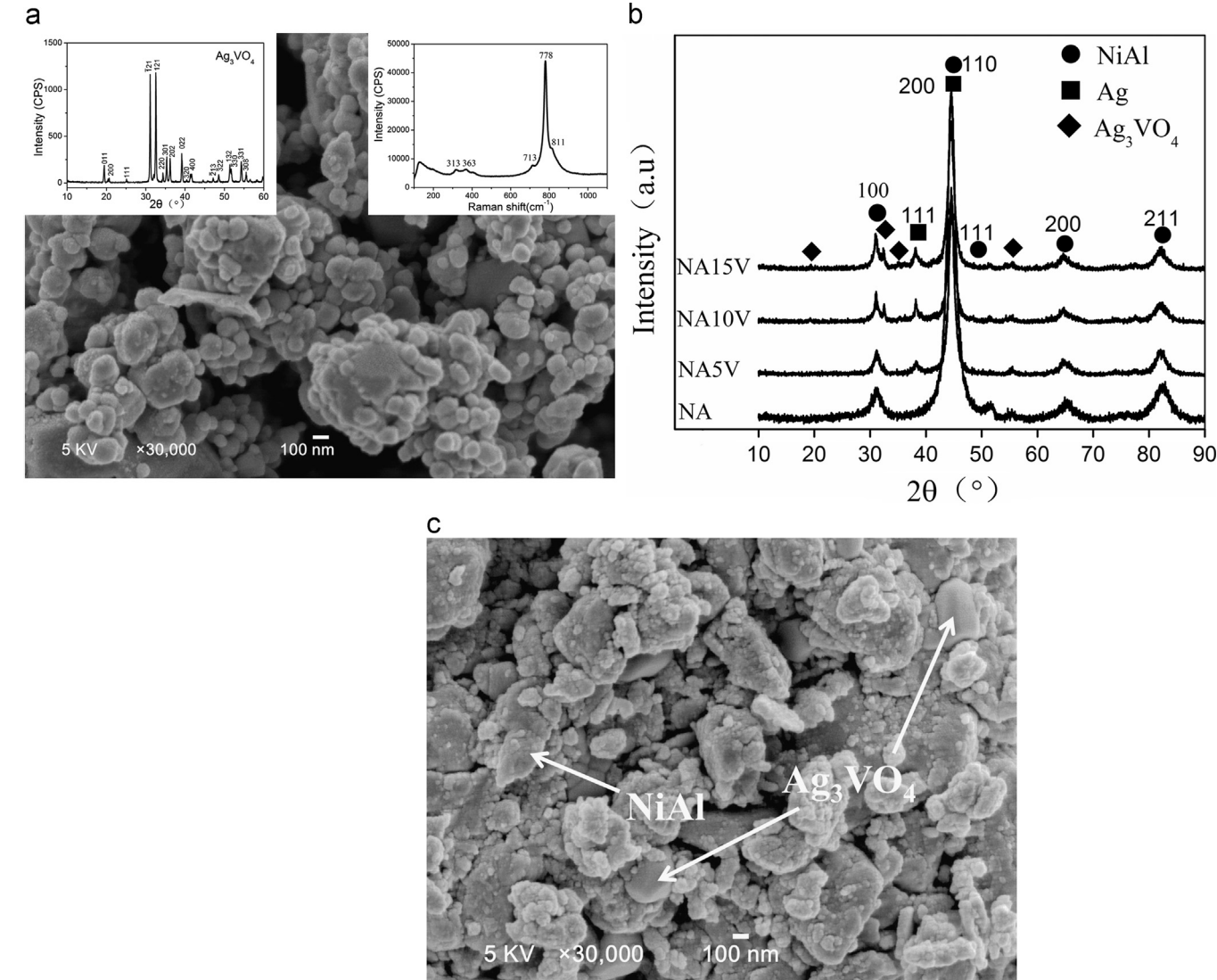


Fig. 1. Characterization of Ag VO nanoparticle (a) X-ray characterization of NiAl-based composites (b) and SEM characterization (c) of milled NA15V composite powders.

to provide a Ni-50 at% Al powders, the contents of Ag_3VO_4 powders were selected to be 0, 5, 10 and 15 wt% (denoted to NA, NA5V, NA10V and NA15V, respectively). Ni-50 at% Al intermetallic were synthesized by mechanical alloying in Fritsch Pulverisette P5 planetary ball, and then the Ag_3VO_4 powders with average grain size about 100 nm were added into the mixed powder for further 5 h ball milling. After milling, the mixed powders were enclosed in a graphite die and sintered under a pressure of 20 MPa in a vacuum hot-pressing furnace at 1300 °C for 60 min, followed by furnace cooling (ZT-45-20, Shanghai Chen Hua Electric Furnace Corp Ltd., China). After sintering, the relative densities of sintered composites were measured by the Archimedes method and microhardness was measured by MH-5-VM microhardness tester equipped with a Vickers diamond pyramid indenter. For each sample, at least ten measurements were carried out using a normal load of 300 g and a dwell time of 5 s, the average value was shown in Table 1.

2.3. Characterization

The composition and crystal structure of milled powders and sintered composites was performed on Philips X' Pert-MRD X-ray diffractometer (XRD) with Cu K α radiation and 40 kV operating voltage

in the 2θ range of 10–80°. Scanning electron microscope (SEM) and transmission electron microscopy (TEM) was utilized to obtain the microstructure. The TEM foils were prepared by conventional procedures that involved mechanical polishing of the cut discs with a 3.0 mm diameter and 30 μm thicknesses, and then electropolished by a Twin-jet thinning in a 5% solution of perchloric acid in methanol at 45 V and –20 °C.

The tribological properties of NiAl-based composites were tested by pin-on-disk tribometer under ambient conditions at different temperatures (RT, 300 °C, 500 °C, 700 °C, and 900 °C, respectively). The pin samples with a size of $\varnothing 5 \text{ mm} \times 15 \text{ mm}$ were made of sintered NiAl-based nanocomposites, one tip of pin was prepared to $\varnothing 5 \text{ mm}$ hemisphere. The disk samples with a size of $\varnothing 24 \text{ mm} \times 8 \text{ mm}$ were made of Inconel 718 alloy. Before test, the pin and disk specimens were polished by 800 grit emery paper and ultrasonically cleaned in an acetone bath. During wear testing, the upper pin specimen was fixed, while the lower disk specimens were rotated at 300 rpm (0.287 m/s) for 1017 m sliding distance (60 min) at 2 N load. The friction torque was continuously measured using a strain-gauge transducer. After wear testing, the specimens were cooled to room temperature and the worn surface was cleaned using compressed N_2 gas. The morphologies and

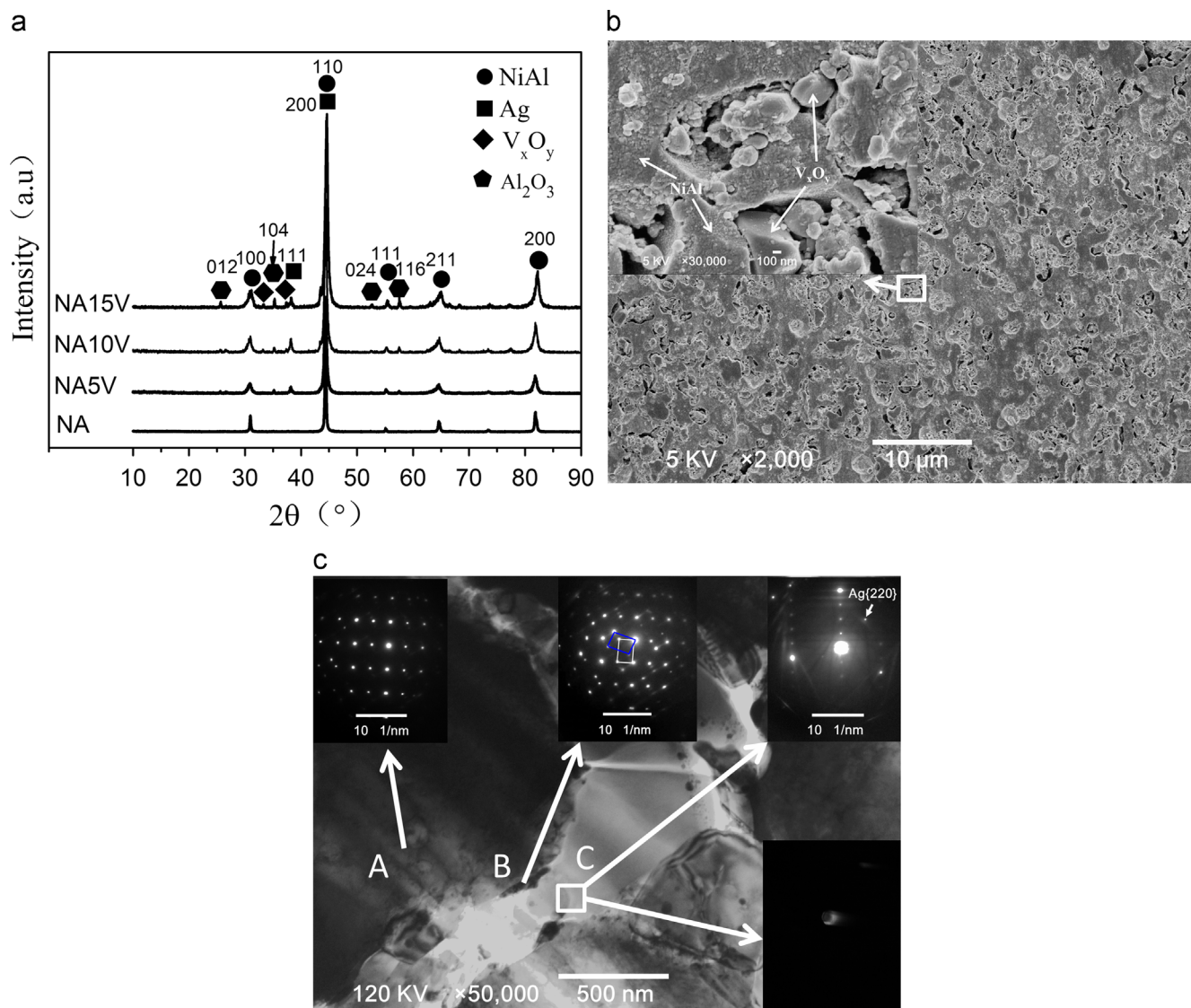


Fig. 2. X-ray diffraction patterns (a) of sintered NiAl-based composites, SEM (b) and TEM (b) characterization of sintered NA15V composite.

phase structure of worn surface were observed by SEM and Renishaw's inVia Micro-Raman using a 633 nm wavelength laser light, respectively [21].

3. Results and discussions

3.1. Material characterization

The crystal structure and microstructure of silver vanadate (Ag_3VO_4) and NiAl/ Ag_3VO_4 powders are presented in Fig. 1. SEM image shows that the precipitates are consisted of spherical particles with diameters in the range from 100 nm to 200 nm, the inset XRD and Raman spectrum suggests that the precipitates are composed of Ag_3VO_4 phase (JCPDS: 19-1154). After milling, XRD patterns indicate that the mixed powders are consisted of NiAl phase (JCPDS: 44-1188), Ag_3VO_4 phase and metallic Ag phase (JCPDS: 04-0783). NiAl powders are synthesized by solid-state diffusion during milling process and the detail process has been described in our previous works [19,22]. The intensities of Ag_3VO_4 peaks increase with the addition of Ag_3VO_4 contents and the appearance of metallic Ag diffraction peaks indicate that Ag_3VO_4 nanoparticles are partly decomposed during milling process. Furthermore, the morphologies of NA15V milled powders indicate that the mixture powders are composed of nanocrystalline NiAl phase with a particle size about 30 nm and Ag_3VO_4 phase with a size of 100–200 nm (Fig. 1c). Thus, XRD and SEM results show that nanocrystalline NiAl/ Ag_3VO_4 powders are successfully synthesized by mechanical alloying technique.

Fig. 2 shows the typical XRD pattern and microstructure of NiAl-based composites after sintering at 1300 °C. The XRD pattern (Fig. 2a) indicates that sintered composites are mainly consisted of B2-ordered NiAl phase, which is consistent with the mixed powders. Nevertheless, the diffraction peaks of Ag_3VO_4 are disappeared and new peaks of metallic Ag, aluminum oxide (Al_2O_3), and vanadium oxide (V_xO_y) are evident detected, suggesting that Ag_3VO_4 phases are completely decomposed during sintering process. The microstructure of NiAl-based composite with 15 wt % Ag_3VO_4 addition (NA15V) is further characterized by SEM and TEM, as shown in Fig. 2b and Fig. 2c, respectively. Combined with the XRD results, it can be found that the sintered composites are consisted of a continuous NiAl phase and homogeneous precipitates (V_xO_y) phase (Fig. 2b). The continuous NiAl phase is densely, but the V_xO_y particles are surrounded by numerous of pores, which is harmful for the mechanical properties of NiAl-based composites. High magnification SEM micrograph (the inset of Fig. 2b) shows that the crystallite size of NiAl phase is about 100 nm and second V_xO_y phase is about 300–500 nm. Furthermore, the crystal structure and microstructure of NA15V composite are also confirmed by TEM (Fig. 2c). The bright-field image indicates that the NA15V nanocomposite is consisted of dark gray phase (area A), bright second phase (area B), small amount of black phase (area C), and a dark area in the bright second phase. Subsequently, selected area diffraction (SAD) pattern confirms that the gray phase (area A) is NiAl matrix phase, black phase (area C) is aluminum oxide precipitates, respectively. Dark-field image and its corresponding SAD pattern confirm that the bright second phase (area B) and inset dark area is vanadium oxide and metallic Ag phase, respectively. The results of TEM are consistent with the XRD and SEM results, thus, it can be deduced that nanocrystalline NiAl-based composites are synthesized by hot-pressing sintering technique.

3.2. Mechanical properties and tribological properties

Table 1 presents the density and microhardness of NiAl-based composites with different contents of Ag VO. The density of

sintered NiAl-based composites increases with the addition of Ag_3VO_4 lubricant, which can be attributed to the existence of high density metallic Ag. Furthermore, the microhardness of sintered NiAl-based composites decreases obviously with increasing of Ag_3VO_4 contents, because metallic Ag and V_xO_y particles are softer than NiAl matrix phase [23]. Furthermore, V_xO_y precipitates are surrounding by numerous of pores, which are significantly deteriorated the mechanical properties of NiAl-based composites.

The friction coefficients and wear rates of NiAl-based composites are shown in Fig. 3. It can be seen that the friction coefficient of NA composite decreases first and then increases with increasing temperature, but the friction coefficient of NiAl-based composite with the addition of Ag_3VO_4 decreases continuously except NA5V composite at 900 °C. For example, the room temperature friction coefficient of NA composite is 0.52 and NA15V composite is 0.31, and the friction coefficient at 900 °C of NA composite is higher to 0.69 and NA15V composite is lower to 0.06. The results show that silver vanadate is an excellent lubricant for NiAl intermetallic, the friction coefficient of NiAl-based composites decreases with the addition of silver vanadate. Furthermore, wear testing results indicate that the wear rate of all NiAl-based composites increase with increasing temperature except NA10V and NA15V composite exhibits a significantly decrease above 700 °C. At RT–500 °C, the wear rate of NA composite is lower than NA10V and NA15V composite, because the wear rate is related to the Microhardness of composite. However, the wear rate of NA10V and NA15V composite decreases obviously above 500 °C, which can be attributed the lubricating effect of silver vanadate. Thus, it can be deduced that Ag_3VO_4 lubricants are beneficial for the improvement of the tribological properties of NiAl intermetallic, especially at elevated temperatures.

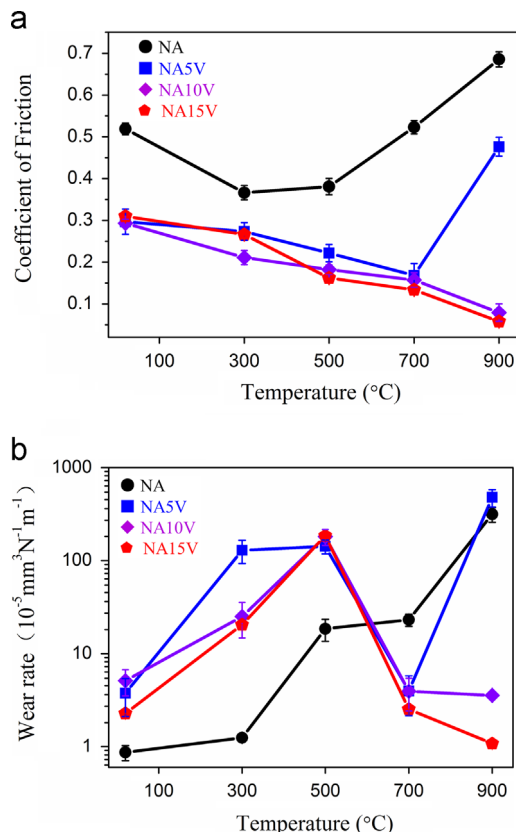


Fig. 3. Friction coefficient (a) and wear rate (b) of NiAl-based composites after wear test at different temperatures.

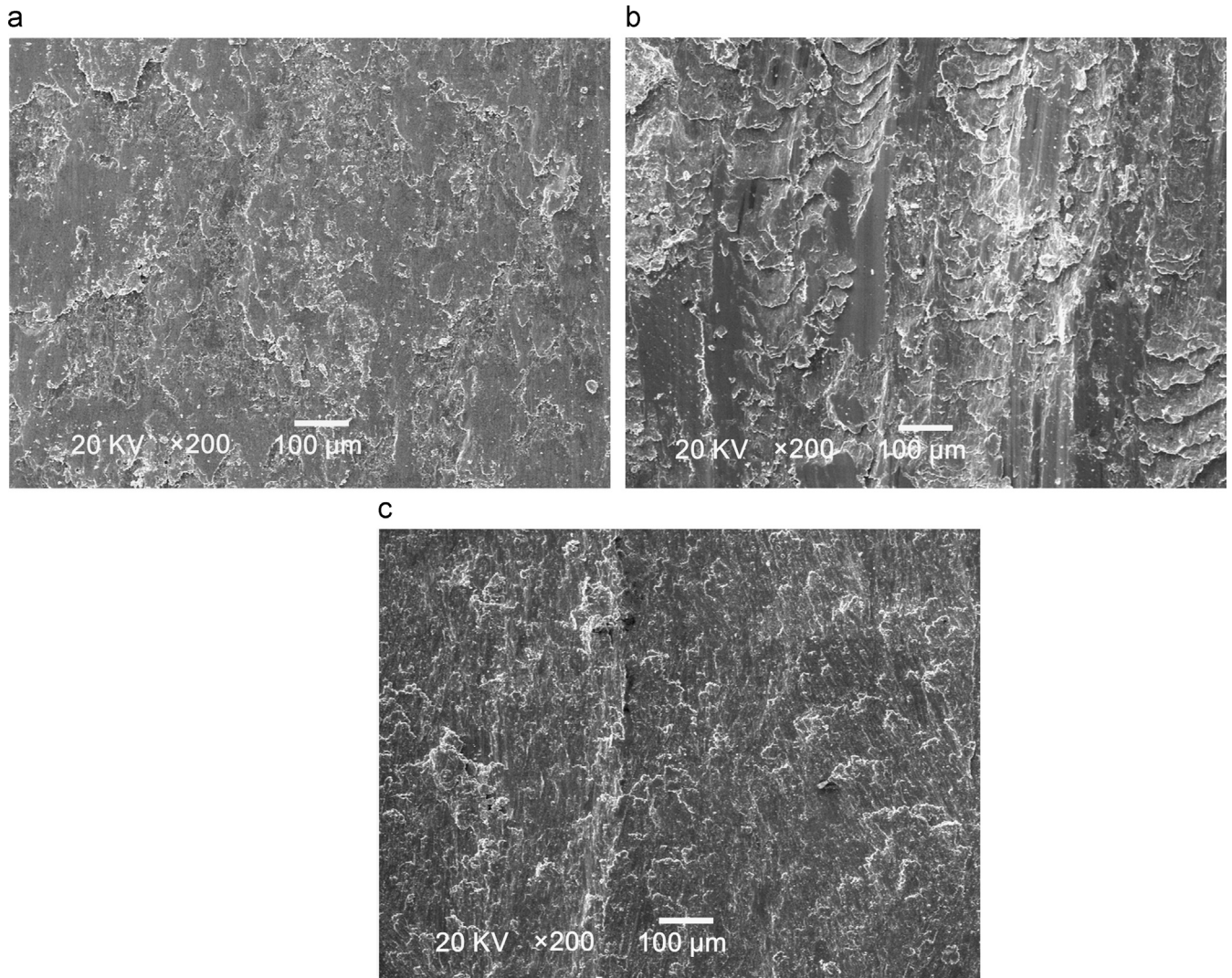


Fig. 4. Worn surface morphologies of NA composite after wear test at different temperatures (a) Room temperature (b) 500 °C (c) 900 °C.

3.3. Worn surface morphologies

The worn surface morphologies of NA composite after wear testing at different temperature are shown in Fig. 4. At room temperature, larger amount of flakes and delamination pits are found on the worn surface, indicating that the worn mechanism is dominated by adhesive wear. Adhesive wear is the result of the direct contact of pin and disk, thus, the shearing of adhesive junctions produces wear particles and flakes when relative motion takes place [24]. At 500 °C, it can be observed that some plastic yielding parallel to the frictional direction, suggesting that the wear mechanism is plastic deformation and micro-cutting. At 900 °C, fracture and serious adhesive wear is the main wear mechanism. The addition of suitable lubricants is an effective way to minimize the points of adhesive wear [25].

The worn surface morphologies of NA15V composite are presented in Fig. 5. It can be seen that the addition of silver vanadate obviously changes the worn morphologies. At room temperature, shallow grooves and some wear debris can be seen on the worn surface, suggesting that the wear mechanism is dominant by microploughing. The addition of silver lubricants indeed minimizes the points of adhesive wear. At 500 °C, the worn surface is covered by a glaze layers together with some wear debris (Fig. 5b), implying that the main wear mechanism is plastic deformation and abrasive wear. At 900 °C, a whole scale glaze layers are formed

on the worn surface, indicating that the wear mechanism is dominated by plastic deformation. It is well known that metal materials often exhibit high ductility at elevated temperatures, which is benefited for the formation of glaze layers. Thus, the smooth and complete glaze layers can be easily formed by the soft debris and the formation of glaze layers is benefitted for the improvement of the tribological properties of NiAl-based composites.

3.4. Phase composition of the worn surface

The phase composition of the glaze layers on the worn surface of NA15V composite after wear testing at 900 °C are characterized by Micro-Raman analyzer, and the results are shown in Fig. 6a. By comparing the Raman spectrum of the glaze layers and un-tested areas, it can be found that the glaze layers are composed of silver vanadate, iron oxides, and nickel oxides, but the un-tested areas are only composed of nickel oxides and aluminum oxide. The Raman results indicate that silver vanadate and iron oxides are formed by the tribo-reaction during the rubbing process, silver vanadate is reproduced by the tribo-chemical reaction of metallic Ag and vanadium oxide, the nickel oxide is formed by oxidation of metallic nickel, and the iron oxide is transferred from the counterpart Inconel 718 disk. It can be deduced that the rubbing process promotes the formation of oxides layers at elevated temperatures, and the

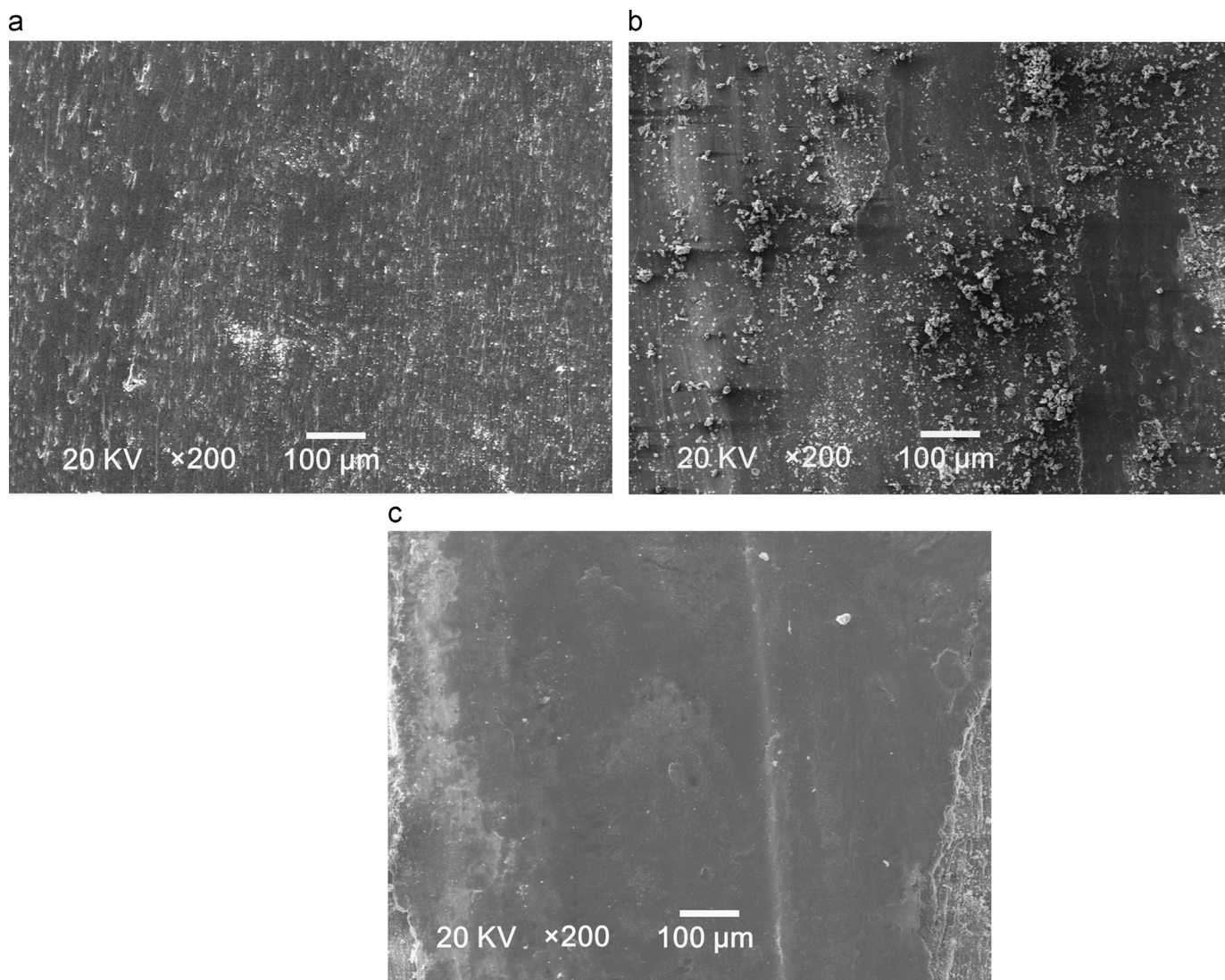


Fig. 5. Worn surface morphologies of NA15V composite after wear test at different temperatures (a) Room temperature (b) 500 °C (c) 900 °C.

formation of glaze layer is remarkable improved the tribological properties of NiAl-based composites at elevated temperatures.

Fig. 6b shows the phase composition of NA15V composite after wear testing. At 500 °C, Raman spectrum of NA15V composite indicates that the glaze layers on the worn surface are primarily consisted of Ag_3VO_4 and AgVO_3 , demonstrating that silver vanadate is reproduced by the tribo-reaction of metallic Ag and V_xO_y during wear testing. At 700 °C, Raman peaks of Ag_3VO_4 are almost disappeared but intensity of AgVO_3 peaks increase obviously, which is due to the fact that AgVO_3 is more stable than Ag_3VO_4 at high temperatures [16]. At 900 °C, Raman results show that the worn surface is mainly consisted of AgVO_3 , Fe_2O_3 , and NiO phase. Thus, it can be deduced that the formation of oxide and reproduction of silver vanadate lubricants are responsible for the decrease of friction coefficient and wear rate at high temperatures.

Fig. 7 shows the worn morphologies of Inconel 718 alloy disk against NA and NA15V composite after wear test at 700 °C and 900 °C, respectively. It can be seen that the worn surface are covered by larger amount of wear debris, and the particles size of wear debris increases with increasing temperature. Combined with the worn surface morphologies of pin sample at same temperatures, it can be concluded that the decrease of the mechanical properties of NiAl matrix phase at high temperature induces the adhesive wear more seriously [2,26]. Thus, addition of

lubricants or improvement the high temperatures mechanical properties are the effective way to improve the adhesive wear action of NiAl intermetallic [27]. In this study, silver vanadate lubricant is selected for NiAl-based composite. The worn surface morphologies of Inconel 718 disk show that a discontinuous glaze layers are formed, which is better to minimize the adhesive wear action. Furthermore, the worn surface morphologies of NA15V pin sample at high temperature also show the main wear mechanism is plastic deformation, indicating that the adhesive wear action of NiAl intermetallic is significantly improved by the addition of suitable lubricants. Subsequently, the phase composition of the worn surface of Inconel 718 disk tested against NA15V composite at 700 °C and 900 °C are presented in Fig. 8. At 700 °C, Raman results indicate that the worn surface are mainly consisted of iron oxide and silver vanadate (Ag_3VO_4), but it changes to silver vanadate (AgVO_3) when temperature increases to 900 °C. The phase composition of Inconel 718 disk is consistent with NA15V pin sample, indicating that AgVO_3 is more stable than Ag_3VO_4 at elevated temperatures.

Fig. 9 shows the composition of the worn surface of NA15V composite at different temperatures. It can be seen that the phase composition automatically changes with increasing temperature, which is benefitted for the decrease of friction coefficient and wear rate of NiAl-based composites. First, metallic Ag is the main

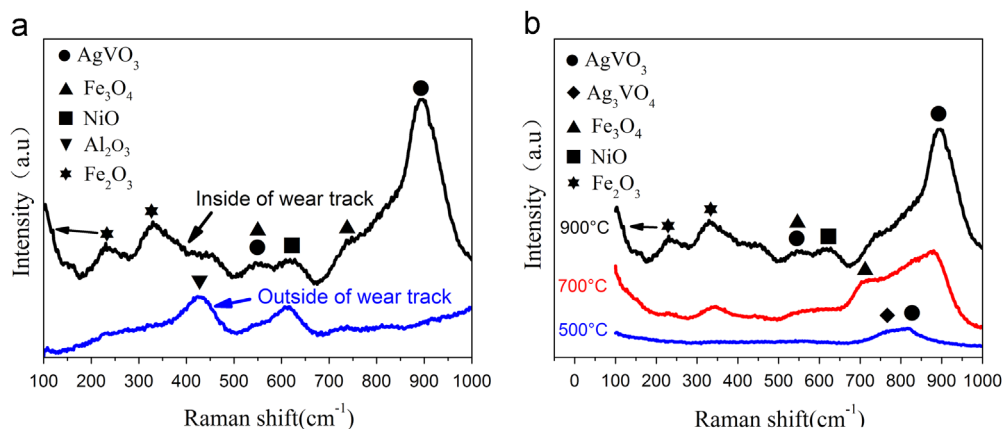


Fig. 6. Raman spectra of the worn surfaces of NA15V composite after wear test. Different area at 900 °C (a) different temperatures of 500 °C, 700 °C, 900 °C, respectively (b).

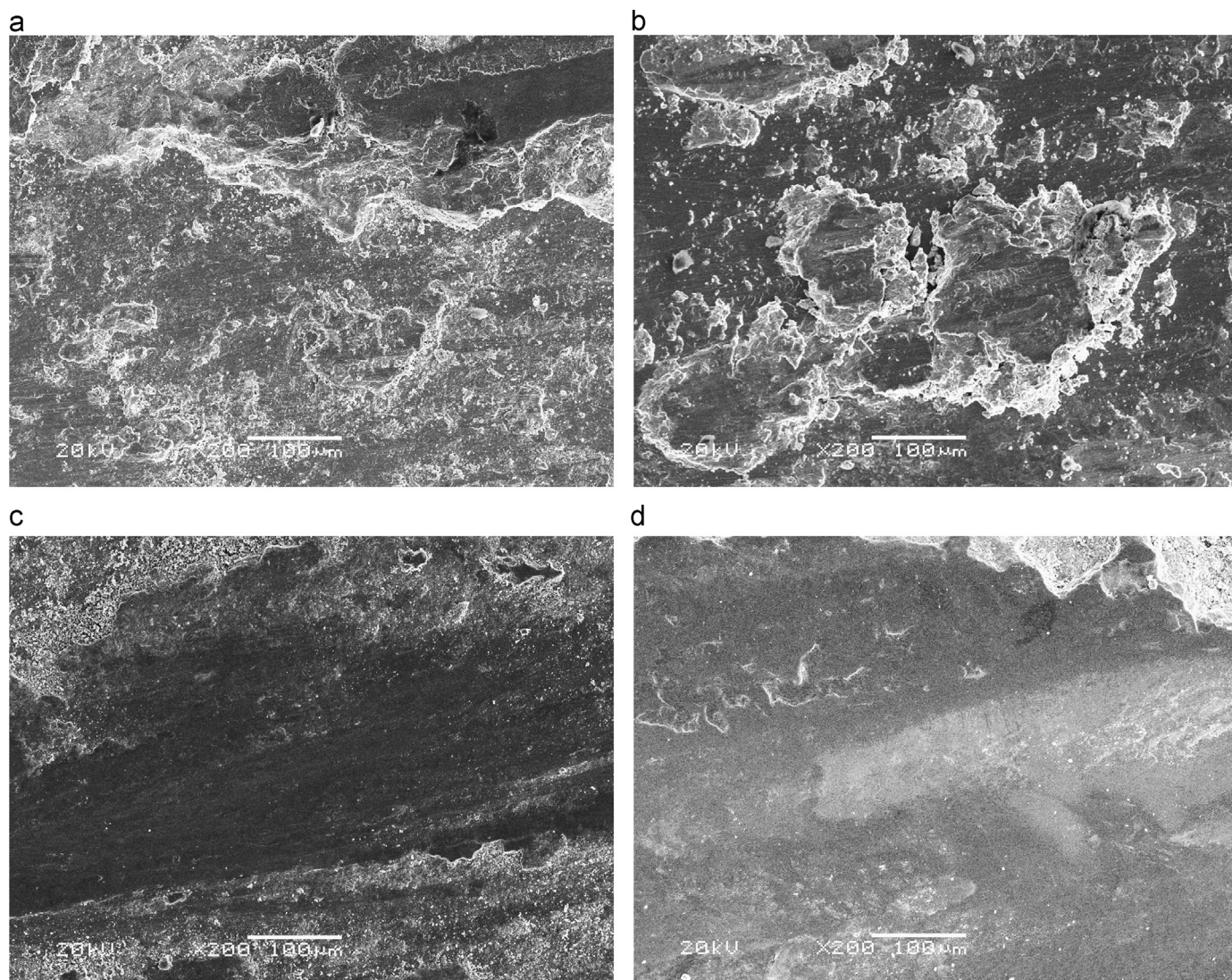


Fig. 7. Worn surface morphologies of Inconel 718 disk against NA at 700 °C (a) 900 °C (b) and NA15V composites at 700 °C (a) 900 °C (b) respectively.

lubricants of room temperatures and lower temperature, which is easily to form a lubricating film during wear testing. At mid-high temperature, oxidation of materials is unavoidable, thus, the formation of iron oxide and Ag_3VO_4 are responsible for the improvement of the tribological properties, and especially the

lamellar structure Ag_3VO_4 is main lubricant. Finally, Ag_3VO_4 is almost disappeared but AgVO_3 is the primary lubricant at elevated temperatures, which is significantly decrease the friction coefficient and wear rate of NiAl-based composites. Thus, it can be deduced that the worn surface of NiAl-based composites is all

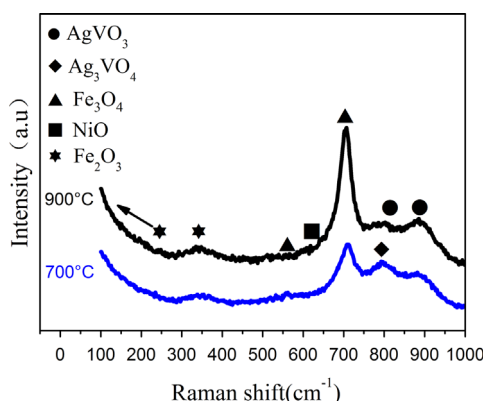


Fig. 8. Raman spectra of the worn surfaces of Inconel 718 disk against NA15V composite at 700 °C, 900 °C, respectively.

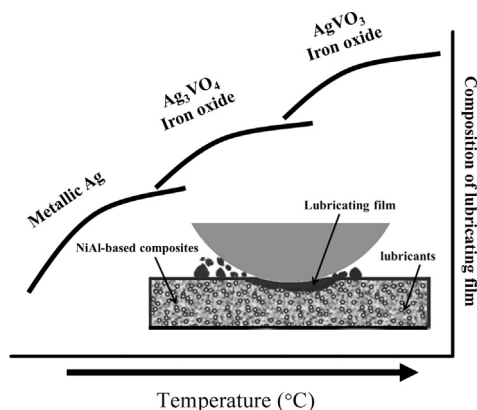


Fig. 9. Schematic of the temperature-adaptive action of NA15V composite at different temperatures.

composed of suitable lubricants at different temperatures. The temperature-adaptive action of the phase composition on the worn surface remarkably improves the tribological behavior of NiAl-based composites at a board temperature range.

4. Conclusions

In this article, silver vanadate nanoparticles were successfully synthesized by hydrothermal method and NiAl/Ag₃VO₄ composites were prepared by mechanical alloying and hot-pressing sintering technique. The microstructures of sintered composites were investigated and the tribological properties of NiAl-based composites were studied against Inconel 718 alloy disk from room temperature to 900 °C. The main conclusions can be drawn as follows:

1. Sintered NiAl-based composites were consisted of B2 ordered NiAl matrix, metallic Ag, and vanadium oxide precipitates, which were distributed homogeneously in the nanocrystalline NiAl matrix. The density NiAl-based composite increased but the Microhardness decreased with increasing Ag₃VO₄ contents, which can be attributed to the formation of high density soft metallic Ag.
2. Wear testing results showed that NiAl/Ag₃VO₄ composites have excellent high temperature tribological properties. The excellent tribological properties (friction coefficient (0.06) and wear

rate ($1.08 \times 10^{-5} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$)) were obtained at 900 °C owing to the reproduction of silver vanadate lubricant.

3. It was proposed that metallic Ag, Ag₃VO₄ and AgVO₃ are the main lubricants of NiAl-based composites at different temperatures, indicating that the temperature adaptive action of the phase composition remarkably improved the tribological properties of NiAl-based composites.

Acknowledgements

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